FIRST QUARTERLY REPORT

For

PRODUCTION OF UNIFORM NICKEL-CADMIUM BATTERY PLATE MATERIALS

(June 13, 1969 to September 12, 1969)

Contract No.: NAS 5-21045

Submitted By

GULTON INDUSTRIES, INC.
Battery & Power Sources Division
Metuchen, New Jersey 08840

For

GODDARD SPACE FLIGHT CENTER
Greenbelt, Maryland

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Goddard Space Flight Center

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ABSTRACT

Sample lots of sintered nickel plaque were prepared in a battery plate production facility. This was done in an experiment to determine the effect of material and process variables on their physical characteristics. The variables studied were: sintering time, sintering temperature, slurry viscosity, sintering atmosphere, priming of substrate, and bulk density of nickel powder.

Batch to batch variation in the nickel powder would also be an observable response. Initial evaluation was concentrated on determining trends caused by the aforementioned variables on porosity and resistivity.

I. INTRODUCTION

The objective of this program is to scale up a laboratory process to a production process for the manufacture of uniform and reliable nickel-cadmium battery plate materials for long term aerospace missions. The specific tasks include a study and evaluation of the effect of material and process variables on the uniformity and characteristics of sintered plaques and impregnated plates.

The required lots of sintered plaques and plates were produced in Gulton Industries' battery plate facility in Gananoque, Ontario. This facility is designed to produce battery plates by the continuous slurry technique. Evaluation and testing of the plaques and plates was made in our Metuchen laboratories. The objective is to determine the necessary process and control procedures under which battery plates of high quality and uniformity can be made reproducibly.

The initial experiments were designed to examine all possible variables which may have an influence on the final product. Particular attention is focused on factors which contribute to non-uniformity.

II. EXPERIMENTAL METHODS AND DATA

A. PROGRAM PLAN

In the initial sintering experiments, it was felt desirable to examine all the variables which have an influence on the pertinent characteristics of sintered nickel plaques. Consequently, a program plan was established where the effect of each of six variables on the quality and uniformity of sintered plaques and impregnated battery plates was examined. The variables selected were:

1. Sintering temperature, at 3 levels.
2. Sintering time (rate of travel of coated strip through the kiln), at 5 levels.
3. Slurry viscosity, at 3 levels.
4. Sintering atmosphere, at 2 levels.
5. Bulk density of nickel powder, at 2 levels, and
6. Priming of nickel substrate prior to application of the slurry coating, at 2 levels.

Varying the drying temperature (and rate) after coating, and prior to sintering, was also considered. However, it was felt that this parameter would have little, if any, effect on the plaque characteristics and was, therefore, omitted from these studies.

The experiments chosen and the particular variables studied in each experiment are listed in Table I. These experiments, performed in the order shown in Table I, should also yield information on the reproducibility of plaques from batch to batch.

TABLE I. TABULATION OF SINTERING EXPERIMENTS

EXPERI-MENT	SLURRY BATCH NO.	SLURRY VISCOSITY cps	PRIMER USED	REDUCING ATMOSPHERE	SINTERING TEMP., °C	SINTERING TIME, Min.
A	1*	70,000	Yes	EXO	900	30
B	"	79,000	"	"	900	20
C	"	75,000	"	"	950	20
D	"	72,000	"	"	1000	20
F	"	76,000	"	"	1000	10
G	"	68,000	"	"	1000	5
H	"	68,000	"	"	1000	2.5
I	"	142,000	"	"	1000	10
J	"	102,000	"	"	1000	10
K	2*	123,000	"	"	1000	10
L	"	125,000	No	"	1000	10
M	"	112,000	"	"	1000	10
N	"	135,000	Yes	"	1000	10
O	"	76,000	"	"	1000	10
P	"	76,000	No	"	1000	10
Q	"	76,000	"	Forming Gas	1000	10
R	"	75,000	Yes	"	1000	10
S	3**	142,000	"	EXO	1000	10
T	"	109,000	"	"	1000	10
U	"	79,000	"	"	1000	10
X	4*	144,000	"	"	950	20
Y	"	144,000	"	"	1000	20
Z	"	144,000	"	"	1000	10

* Nickel Powder, Lot No. B/3, Bulk density 0.92 gm/cc
** Nickel Powder, Lot No. B/998, Bulk density 0.85 gm/cc

B. SLURRY PREPARATION

Four batches of nickel slurry were prepared for the sintering experiments. These consisted of the following:

B1 & B2	-	180 lbs. nickel powder #287, Lot B/3
		180 lbs. water
		4 lbs. Methocell
B3	-	180 lbs. nickel powder #287, Lot B/998
		180 lbs. water
		4 lbs. Methocell
B4	-	180 lbs. nickel powder #287, Lot B/3
		170 lbs. water
		4 lbs. Methocell

The bulk densities of the nickel powders were determined using a Scott Volumeter. Two powder samples were taken from each of the four barrels used, one from the top and one from the bottom, to check uniformity within the same barrel and from one barrel to the next within the same lot.

The values shown in Table II do show a slight variation between top and bottom of the barrels, with the bottom somewhat more dense. This is most likely due to settling of the smaller particles during handling and shipment. The bulk densities of B1, B2 and B4 should be identical, as they have the same lot number. While B1 and B4 are in essential agreement with the manufacturer's value, there is a discrepancy between the measured and reported values for B2 and B3.

TABLE II. - BULK DENSITY OF NICKEL POWDERS

POWDER SAMPLE	MEASURED DENSITY *	
	gm/cu in.	gm/cc
1. B1, Top, Lot B/3	15.2239	0.9288
2. B1, Bottom, Lot B/3	15.2694	0.9316
3. B2, Top, Lot B/3	15.7275	0.9595
4. B2, Bottom, Lot B/3	15.8883	0.9693
5. B3, Top, Lot B/998	13.2938	0.8110
6. B3, Bottom, Lot B/998	13.4074	0.8180
7. B4, Top, Lot B/3	15.1394	0.9236
8. B4, Bottom, Lot B/3	15.3914	0.9390

* Bulk densities reported by the manufacturer were 0.92 gm/cc and 0.85 gm/cc for Lots B/3 and B/998 respectively.

Deionized water, at 70°C, was added to wet the powders, and rolled for 10 hrs. in polyethylene lined vessels to uniformly disperse the binder in the nickel powder. The slurry was allowed to stand in a water cooled bath for 14-16 hours to affect dissolution of the binder, "poled"** to adjust its viscosity by the addition of water, and transferred to the feeder for the sintering experiments. The viscosity of the slurry was checked with a Brookfield Viscometer at 2 rpm using a #5 spindle. The initial viscosities of the four slurry batches prepared above were:

B1 - 130,000 centipoises	B3 - 142,000 centipoises
B2 - 130,000 "	B4 - 152,000 "

** "Poling" is a method of mixing without introducing air bubbles in the slurry.

C. SINTERING EXPERIMENTS

The sintering experiments were carried out in the order shown in Table I. Grade "A" perforated nickel foil, 0.003 in. thick and 7-1/2 in. wide, was used as the substrate. Where a change in firing temperature was involved in going from one experiment to the next, sufficient time (about 30 minutes) was allowed for the kiln to come to equilibrium. The sintering time was controlled by adjustment of the rate at which the coated nickel substrate travelled through the sintering oven. To change the slurry, all vessels were emptied and cleaned before introducing the new material (either of a different viscosity or a different batch).

Approximately fifty feet of sintered plaque material was run under each of the experimental conditions listed above. The kiln atmosphere was EXO gas* in each case, excepting experiments Q and R, where Forming Gas (10% H_2, 90% N_2) was used to determine whether the composition of the reducing atmosphere bears an influence on the sintered nickel.

The firing temperatures in the sintering kiln, as indicated by direct readout thermocouples, are shown in Table III.

Samples of slurry were collected for determination of solids content. This was done by first driving off the water in a vacuum oven at about 2 psia and 100°C. The slurry sample was left in the vacuum oven overnight, cooled and weighed. This process was repeated two or three times until no change in weight was noted. The dried samples are then further treated to burn off the binder to determine the nickel content. At this writing, this work is incomplete and the results of these experiments will be reported later.

Following the sintering, the plaque was coined and cut into strips of about three feet in length. Each strip was numbered to enable us to identify its exact position. The strips were divided into three groups in such a manner that each group was identically representative of the full run.

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* 17% H_2, 83% N_2

TABLE III. - SINTERING KILN TEMPERATURES

EXPERIMENT	TEMPERATURE, °F		
	BOTTOM	CENTER	TOP
A	1640	1660	1640
B	1620	1660	1650
C	1735	1740	1730
D	1830	1840	1830
F	1820	1835	1820
G	1820	1835	1820
H	1830	1840	1810
I	1830	1840	1830
J	1830	1840	1830
K	1830	1840	1830
L	1825	1840	1840
M	1830	1840	1830
N	1830	1840	1830
O	1830	1840	1840
P	1830	1840	1830
Q	1830	1840	1830
R	1830	1840	1830
S	1820	1835	1820
T	1830	1835	1820
U	1830	1835	1820
X	1735	1740	1730
Y	1830	1840	1830
Z	1830	1840	1830

Two groups were impregnated, converted to $Ni(OH)_2$ and $Cd(OH)_2$, and set aside for a subsequent determination of weight gain and active material distribution.

The third group was blanked into individual plates (2.75" x 5.90") for characterization as plaque material.

D. POROSITY MEASUREMENTS

Porosity measurements were made using 2 inch square samples of plaque material. The test specimen was weighed dry, and then reweighed while suspended in a liquid (kerosene). Some time (2-3 minutes) was allowed for the liquid to penetrate the pores before making the weight measurement. The saturated specimen was removed from the kerosene and again weighed after the excess liquid had been removed from the surface using a slightly moistened piece of cotton cloth, being careful not to withdraw any liquid from the pores.

The porosity of the test sample was calculated using the formula:

$$P = \frac{W - D}{W - S} \times 100$$

where D = dry weight (before immersion),

S = Suspended weight, and

W = saturated weight in air

Duplicate measurements were made on each experimental run with the results listed in Table IV.

TABLE IV. POROSITY OF SINTERED PLAQUES

EXPERIMENT AND STRIP NUMBER	THICKNESS (Inches)	POROSITY (%)	EXPERIMENT AND STRIP NUMBER	THICKNESS (Inches)	POROSITY (%)
A-1	.025	72.9	M-10	.031	72.6
A-9	.028	72.8	N-2	.033	72.8
B-1	.027	72.5	N-11	.033	72.6
B-10	.027	73.1	O-1	.032	72.7
C-2	.028	71.9	O-9	.031	73.6
C-11	.028	71.5	P-2	.030	72.4
D-2	.027	73.7	P-11	.032	73.5
D-11	.027	73.0	Q-1	.031	72.4
F-1	.030	73.8	Q-9	.032	72.5
F-11	.031	73.7	R-1	.033	73.5
G-2	.033	74.8	R-10	.032	72.9
G-11	.034	74.2	S-2	.034	73.2
H-1	.034	74.3	S-11	.034	73.6
H-10	.035	74.9	T-1	.033	73.8
I-1	.032	74.0	T-10	.034	74.1
I-10	.031	73.5	U-1	.033	73.9
J-2	.032	71.7	U-10	.034	73.3
J-11	.033	72.4	X-1	.033	72.6
K-1	.034	73.9	X-10	.035	72.5
K-10	.035	73.8	Y-1	.033	72.1
L-1	.034	73.8	Y-10	.032	72.0
L-9	.033	73.1	Z-2	.035	73.2
M-1	.032	72.9	Z-11	.031	73.0

E. RESISTIVITY MEASUREMENTS

To measure the resistivity of the sintered plaques, a device was fabricated where copper bars were attached to opposite sides and across the full width of the test sample. With a measured current flowing through the test sample, the voltage drop between two points, a known distance apart, and parallel to the direction of the current flow, was measured. The voltage probes were spring loaded to maintain a constant contact pressure on the surface of the sinter. The test setup for making these measurements is shown in Figure 1. From the measured voltage drop, the current, and the cross-sectional area of the test sample, the resistivity, ρ, was calculated using the formula:

$$\rho = \frac{EA}{I \ell}$$

where: E = measured voltage drop

A = cross-sectional area

I = current flowing through test sample, and

ℓ = distance between voltage probes

To check out the device, measurements were made on a sample plaque at several different currents ranging from 2 amperes to 20 amperes. At first, the current probes were attached to the plaque substrate. The same measurements were then repeated with the current probes attached to the sinter. The results of these measurements, shown in Table V, indicated that an ohmic relationship exists between current and voltage, and that the results of the measurement were not influenced by whether the current probes were attached to the substrate or the sinter. Further, the current did not cause heating that would result in errors in the determination. Plaque resistivities of all the experimental runs were measured in triplicate; one at the beginning, one in the middle, and one at the end of the run. The results are shown in Table VI.

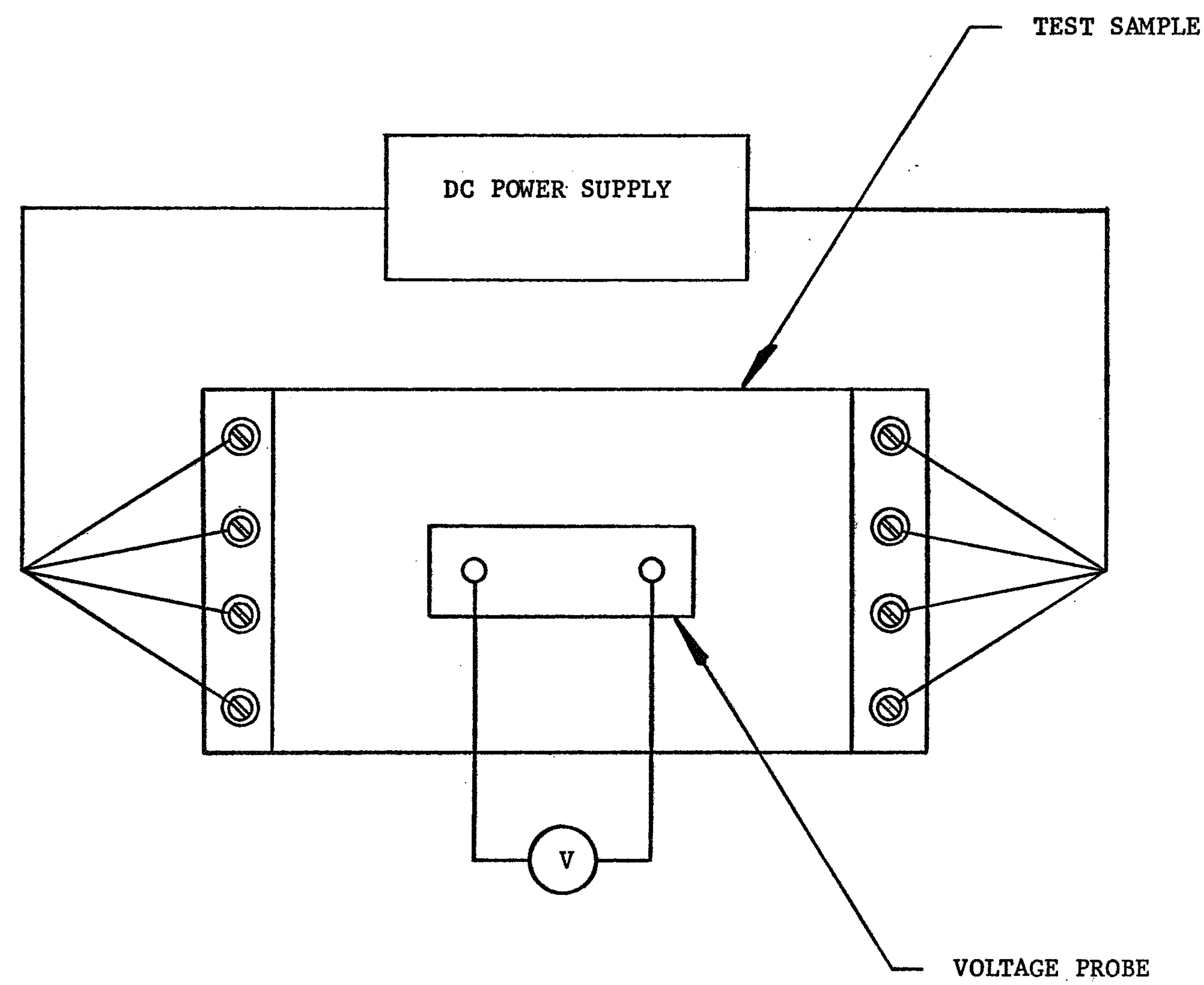

FIGURE 1. TEST SETUP FOR MEASURING PLAQUE RESISTIVITY

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TABLE V.

CURRENT-VOLTAGE RELATIONSHIP, SINTERED NICKEL PLAQUE

CURRENT FLOW THRU TEST SAMPLE	VOLTAGE DROP	
	CONTACT MADE TO SUBSTRATE	CONTACT MADE TO SINTER
20 Amps	17.25 mV	17.50 mV
10 "	8.75 "	8.75 "
8 "	7.00 "	7.00 "
6 "	5.25 "	5.25 "
4 "	3.50 "	3.50 "
2 "	1.75 "	1.75 "

The three values for each of the experimental runs were averaged and listed in Tables VII to XII to show the effect of sintering temperatures, sintering time, slurry viscosity, primer, sintering atmosphere, and batch number (reproducibility) on plaque resistivity.

TABLE VI - RESISTIVITY OF SINTERED NICKEL PLAQUES *

IDENTIFICATION	RESISTIVITY, ohm-cm x 10^{-5}	IDENTIFICATION	RESISTIVITY ohm-cm x 10^{-5}
A-3	7.2	M-3	8.1
A-11	6.9	M-9	8.1
A-14	7.3	M-18	8.1
B-3	7.3	N-1	8.7
B-8	7.8	N-7	8.9
B-15	7.8	N-13	8.1
C-1	7.7	O-5	8.1
C-10	7.6	O-11	8.1
C-19	8.2	O-20	8.1
D-4	7.6	P-1	7.9
D-10	8.2	P-10	8.1
D-16	7.6	P-19	7.9
F-3	8.8	Q-2	8.1
F-10	7.6	Q-8	7.6
F-22	7.6	Q-14	7.6
G-4	9.0	R-3	8.7
G-16	9.3	R-6	7.8
G-25	9.0	R-12	8.4
H-3	9.5	S-1	8.4
H-12	9.3	S-13	8.6
H-21	9.7	S-19	8.6
I-3	8.4	T-3	8.3
I-9	7.8	T-19	8.6
I-18	8.4	T-18	8.4
J-1	7.9	U-3	7.8
J-10	8.1	U-9	8.1
J-19	8.1	U-18	8.0
K-3	8.7	X-3	7.8
K-12	8.9	X-9	7.6
K-18	8.1	X-15	7.6
L-2	8.4	Y-3	7.1
L-11	8.4	Y-9	7.1
L-17	8.4	Y-15	7.3
		Z-1	8.3
		Z-7	8.0
		Z-13	7.9

* Measurements made at 10 amperes

TABLE VII - RESISTIVITY VS. SINTERING TEMPERATURE

EXPERIMENT	BATCH	VISCOSITY (cps)	SINTERING TEMP./TIME	ρ
B	B_1	70,000	900/20	7.6×10^{-5}
C		75,000	950/20	7.8×10^{-5}
D		72,000	1000/20	7.8×10^{-5}
X	B_4	144,000	950/20	7.7×10^{-5}
Y		144,000	1000/20	7.2×10^{-5}

TABLE VIII. - RESISTIVITY VS. SINTERING TIME

EXPERIMENT	BATCH	VISCOSITY	SINTERING TEMP./TIME	ρ
D	B_1	72,000	1000/20	7.8×10^{-5}
F		76,000	1000/10	8.0×10^{-5}
G		68,000	1000/5	9.1×10^{-5}
H		68,000	1000/2½	9.5×10^{-5}
Y	B_4	144,000	1000/20	7.2×10^{-5}
Z		144,000	1000/10	8.1×10^{-5}

TABLE IX. - RESISTIVITY VS. SLURRY VISCOSITY

EXPERIMENT	BATCH	VISCOSITY	SINTERING TEMP./TIME	ρ
I	B_1	142,000	1000/10	8.2×10^{-5}
J		102,000	1000/10	8.0×10^{-5}
F		76,000	1000/10	8.0×10^{-5}
K	B_2	123,000	1000/10	8.6×10^{-5}
N		135,000	1000/10	8.6×10^{-5}
O		76,000	1000/10	8.1×10^{-5}
S	B_3	142,000	1000/10	8.5×10^{-5}
T		109,000	1000/10	8.4×10^{-5}
U		79,000	1000/10	8.0×10^{-5}

TABLE X. EFFECT OF PRIMER ON RESISTIVITY

EXPERIMENT	BATCH	VISCOSITY	PRIMER	SINTERING TEMP./TIME	ρ
K	B_2	123,000	Yes	1000/10	8.6×10^{-5}
L		125,000	No	1000/10	8.4×10^{-5}
N		135,000	Yes	1000/10	8.6×10^{-5}
M		112,000	No	1000/10	8.1×10^{-5}
O		76,000	Yes	1000/10	8.1×10^{-5}
P		76,000	No	1000/10	8.0×10^{-5}

TABLE XI. RESISTIVITY VS. SINTERING ATMOSPHERE

EXPERIMENT	BATCH	VISCOSITY	PRIMER	ATM.	SINTERING TEMP./TIME	ρ
O	B_2	76,000	Yes	EXO.	1000/10	8.1×10^{-5}
R		75,000	Yes	F.G.	1000/10	8.3×10^{-5}
P		76,000	No	EXO.	1000/10	8.0×10^{-5}
Q		76,000	No	F.G.	1000/10	7.8×10^{-5}

TABLE XII. RESISTIVITY VS. BATCH NUMBER

EXPERIMENT	BATCH	VISCOSITY	SINTERING TEMP./TIME	ρ
I	B_1	142,000	1000/10	8.2×10^{-5}
K	B_2	123,000	1000/10	8.6×10^{-5}
S	B_3	142,000	1000/10	8.5×10^{-5}
Z	B_4	144,000	1000/10	8.1×10^{-5}
U	B_1	102,000	1000/10	8.0×10^{-5}
N	B_2	135,000	1000/10	8.6×10^{-5}
T	B_3	109,000	1000/10	8.4×10^{-5}
F	B_1	76,000	1000/10	8.0×10^{-5}
O	B_2	76,000	1000/10	8.1×10^{-5}
U	B_3	79,000	1000/10	8.0×10^{-5}

III. DISCUSSION

The characterization of the experimental plaques is in progress, hence, the discussion must be limited to trends shown by the results obtained to data.

Perhaps one of the more surprising results obtained is the relatively narrow range of porosities found in these experiments. These values ranged from 71.5% (C-11) to 74.9% (H-10).

The method used to determine porosity has one inherent source of error; namely, removing the excess liquid from the surface before weighing the liquid saturated sample in air.

The degree of sintering is a function of both time and temperature. The highest porosity measured was in the samples fired at 1000°C for the shortest period (2-1/2 minutes). This is not unexpected considering that the firing time is in all probability insufficient to allow strong particle-to-particle bonding to take place.

The lowest porosity was measured on the samples fired at 950°C for 20 minutes, rather than those fired at 1000°C for the same period, as one would expect. No explanation is offered for this observation at this time. The effects of viscosity and batch, as examples, also affect response to experimental variables.

The resistivities of the samples measured ranged from 6.9×10^{-5} ohm-cm to 9.7×10^{-5} ohm-cm, with the lowest value being observed on samples A (900°C, 30 min.). The observed trends were as might be expected. Increasing either the firing time or the firing temperature tended to decrease resistivity. The effect of slurry viscosity on plaque resistivity was not discernible from the number of samples studied. These measurements will be repeated with a larger number of samples to determine any real effects.

The data in Table IX suggest that the effect of the primer is to slightly increase resistivity. Possibly, this material leaves some residue which acts as a resistive barrier between the substrate and the sinter.

The effect of the sintering atmosphere (forming gas vs. EXO) appears negligible. This conclusion, however, should be reaffirmed by a larger sampling.

Batch-to-batch uniformity, as determined by plaque resistivity, appears to be good, as shown by the results in Table XI.

The results thus far indicate that uniformity of plaques in a production facility is, indeed, achievable. It remains to be seen whether improved uniformity is obtained in impregnation, and later, in cell performance. This is the work planned for the next interval.

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